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Citation: Wei, Jiacheng, Saharudin, Mohd, Vo, Thuc and Inam, Fawad (2018) Effects of surfactants on the properties of epoxy/graphene nanocomposites. Journal of Reinforced Plastics and Composites. ISSN 0731-6844

Published by: SAGE

URL: http://journals.sagepub.com/doi/full/10.1177/07316... <http://journals.sagepub.com/doi/full/10.1177/0731684418765369>

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# Effects of surfactants on the properties of epoxy/graphene nanocomposites

**Jiacheng Wei, Mohd Shahneel Saharudin, Thuc Vo and Fawad Inam**

**Abstract**

Recently, graphene has attracted extensive research interests due to its superior mechanical, electrical and thermal performance. Small loadings of graphene can increase the properties of epoxy significantly. However, because of the large surface area of graphene, it is a challenge to disperse graphene in liquid epoxy. Strong van der Waals force causes reaggregation of graphene in the matrix. As commonly used surfactants, sodium dodecyl sulphate and gum arabic have been used a lot to de-bundle graphene, however, their dispersing efficiencies for graphene in epoxy matrix is unknown. Therefore, to evaluate their dispersing efficiencies, epoxy/graphene nanocomposites had been made and mechanical properties, dynamic mechanical analyzer, thermal gravimetric analysis and scanning electron microscopy images of nanocomposites had been tested. The results show that the properties of nanocomposites had been enhanced largely after using sodium dodecyl sulphate and gum arabic. Sodium dodecyl sulphate shows higher dispersing effective-ness than gum arabic.

**Keywords**

Graphene, epoxy, nanocomposites, surfactants, sodium dodecyl sulphate, gum arabic

## Introduction

As a single-layered carbon sheet, graphene has attracted extensive research interest due to its superior mechanical, electrical, and thermal performance.[1,2] Due to its unique structure, both layered and graphitized, graphene shows advantages in the preparation of high properties nanocomposites.[3] With the large surface area and the planar structure, graphene can increase the properties of nanocomposites significantly, which is due to the bulky surface contact of graphene and the polymer.[4]

Epoxy is one of the most adaptable and widely sold high-performance material in the world because of its superlative mechanical properties, thermal stability, solvent resistance, and ease of processing.[5–7] More than that, due to other properties of chemical resistance, low price, epoxy exhibits wide applications,[8–10] epoxy-based materials are used widely in the applications of engineering, construction, and electronics, etc.

Small loadings of graphene can increase the properties of epoxy significantly.[13] However, the premise of this enhancement is the uniform distribution of the filler in the matrix.[14] The maximum surface content of graphene and epoxy can only be achieved at the uniformly dispersed graphene,[15] thus, the filler could share external stress, block the advancing cracks, and then elevate the mechanical properties of nanocomposites.[16] While in opposite, poorly dispersed graphene acts as stress raiser and causes stress concentration,[17] which deteriorates the mechanical properties.[18] However, graphene is unsuited to disperse in liquid matrix due to its large surface area. The strong van der Waals force makes graphene attracts each other, and thus induces the reaggregation of graphene.[19,20]

To solve this problem, surface functionalization of graphene is commonly used.[21–24] Without forming actual chemical bonds, surfactant functionalization connects the graphene and functional groups by only physical

Department of Mechanical and Construction Engineering, Northumbria University, Newcastle upon Tyne, UK

**Corresponding author:**
Fawad Inam, Department of Mechanical and Construction Engineering, Northumbria University, Northumberland Road, Newcastle NE1 8ST, UK.
Email: fawad.inam@northumbria.ac.uk

adsorption, which involves the advantage of easy processability and high de-bundling effectiveness.[25,26] Sodium dodecyl sulphate (SDS) and gum arabic (GA), as widely used amphiphilic water-soluble dispersant, exhibits high efficiencies to de-bundle graphene from aggregates. For SDS, the sulphate groups were negatively charged and able to de-bundle graphene by electrostatic repulsion.[27,28] In regard with GA, it is able to de-bundle graphene by steric repulsion requires only the adsorption of GA chains on the surface of graphene.[29] Therefore, SDS and GA have been widely used to disperse graphene. For example, Amoli et al.[30] prepared an electrically conductive adhesive, by using SDS, a stable graphene dispersion had been achieved and the resultant material showed significant electrical conductivity at noticeably low graphene content. Hajian et al.[31] prepared polyvinyl butyral/graphene nanocomposite by SDS, the prepared nanocomposites showed good toughness and flexibility. Furthermore, SDS has also been reported to prepare graphene nanocomposites in polyvinyl alcohol,[32] polyurethane[33] and polystyrene[34] matrices. For GA, by exfoliating graphite in GA aqueous solution, high yielding and stable dispersion of graphene had been achieved.[35–37] GA has also been reported to disperse graphene and produce hydrogel,[38] polyethylene oxide[39] nanocomposites, etc.

However, although SDS and GA had been widely used to prevent graphene reaggregation, their dispersion effectiveness for graphene is still not yet fully studied. In this work, SDS and GA have been chosen to compare their dispersion effectiveness of graphene in epoxy matrix for the first time. Mechanical properties, dynamic mechanical analyzer (DMA), thermal gravimetric analysis (TGA) and scanning electron microscopy (SEM) images of nanocomposites have been tested to evaluate their dispersing efficiencies.

## Experimental techniques

### Materials

The epoxy system was offered by Polyfibre UK Ltd. This system offers good all-around properties and consists of EPOPHENEHA 57 diamine hardener and EPOPHEN EL5 bisphenol A–based liquid epoxy. Graphene nanoplatelets used in this study were offered by Graphene Laboratories Inc., USA. The average specific surface area of graphene is 80 $m^2/g$ and the average lateral size is 4.5 μm. SDS was purchased from Sigma–Aldrich, product code. 75746. GA was bought from Fisher Scientific, product code. 10165010.

### Sample preparation

As our previous research,[13] 0.3 wt% graphene loading shows the maximum property enhancement to epoxy, therefore, in this work, 0.3 wt% nanocomposites were prepared with different surfactants to compare their dispersing efficiencies.

One set of sample was prepared without any surfactant, market as G-0.3. Firstly, a JPL Ultra7000 bath sonicator with a sonication power of 50 W was used to disperse graphene in the liquid epoxy. Subsequently, the hardener was added by 5 min hand stirring and 5 min bath sonication, and then the entrapped air bubbles were removed by vacuum degassing. Finally, mold casting was carried out to cure the material for 6 h at room temperature and then 6 h at 80°C.

SDS and GA were used to prepare another two sets samples. Firstly, 2.25 g/L SDS/GA aqueous solution was prepared in a beaker by bath sonication, graphene was then added to the beaker and sonicated for 0.5 h. To get rid of the water, the dispersion was heated to 95°C overnight and then obtained the SDS modified graphene and GA-modified graphene. Finally, the same method of G-0.3 samples was applied with the SDS-modified graphene/GA-modified graphene to prepare nanocomposites.

### Characterization

Tensile, flexural and fracture properties were measured by an Instron, 3382 Universal Mechanical Testing Machine. ASTM D638 was referred to test the tensile properties with the specimen thickness of 4mm. ASTM D790 was referred to test the flexural properties with the sample dimension of $70 \times 12.7 \times 3$ mm, and the support span is 48 mm. Fracture toughness ($K_{1C}$) was tested according to ASTM D5045 with the sample dimension of $56 \times 6 \times 3$ mm, the span length is 36 mm and the crack length is 3 mm. Equation (1) was used to calculate the $K_{1C}$

$$K_{1C} = \frac{P_{max} f\left(\frac{a}{w}\right)}{BW^{1/2}} \tag{1}$$

where $f(a/w)$ was calculated using equation (2), $P_{max}$ was taken as the maximum value on the load–displacement curve, $B$ is the sample thickness (mm), $a$ is the length of the crack and kept as and $0.5W$, $W$ is the width of sample (mm). Equation (3) was used to calculate the critical strain energy release rate ($G_{1C}$). In the equation, $E$ is Young's modulus (MPa), $v$ was taken as 0.35 as epoxy's Poisson ratio.

$$f\left(\frac{a}{w}\right) = \frac{\left[\left(2 + \frac{a}{w}\right)\left\{0.0866 + 4.64\left(\frac{a}{w}\right) - 13.32\left(\frac{a}{w}\right)^2 + 14.72\left(\frac{a}{w}\right)^3 - 5.6\left(\frac{a}{w}\right)^4\right\}\right]}{\left(1 - \frac{a}{w}\right)^{3/2}} \tag{2}$$

$$G_{1C} = \frac{K_{1C}{}^2(1 - v^2)}{E} \quad (3)$$

Vickers microhardness was measured by Buehler Micromet II, 10 s with the load of 200 g had been through all samples. Six samples were measured for each set of tests. Figure 1 shows the schematics of the testing samples.

To determine the loss factor tan $\delta$ and the storage modulus ($E'$), a Perkin Elmer – 8000 DMA had been applied to conduct the test. Sample dimensions of $30 \times 8 \times 2.5$mm had been tested by single cantilever method. A thermal gravity analyzer (Q500, TA instrument) was used to carry out the TGA under the nitrogen atmosphere ramp from room temperature to 650°C. To evaluate the fracture modes of the nanocomposites, an FEI Quanta 200 electron microscope had been used to carry out the SEM test. Fourier Transform Infrared Spectroscopy (FTIR) was carried out at room temperature by the FTIR spectrophotometer (Perkin Elmer, L1185247).

## Results and discussion

### *FTIR test*

To evaluate if SDS and GA have been successfully grafted to graphene surface, FTIR of original and SDS/GA-modified graphene have been tested and shown in Figure 2. For original graphene, as graphene constitutes by carbon only, no specific functional group can be seen on the spectrum. For GA–graphene samples, the peaks at 1608.3 $cm^{-1}$ and, 1020 $cm^{-1}$ in the spectra are attributed to stretching vibrations of the C=O and C–O–C structures in the GA, respectively. Another evidence GA is present on the surface of graphene is the wide diffraction peak in the range of 3000–3700 $cm^{-1}$, this features the hydroxyl groups of the polysaccharide, which is the main composition

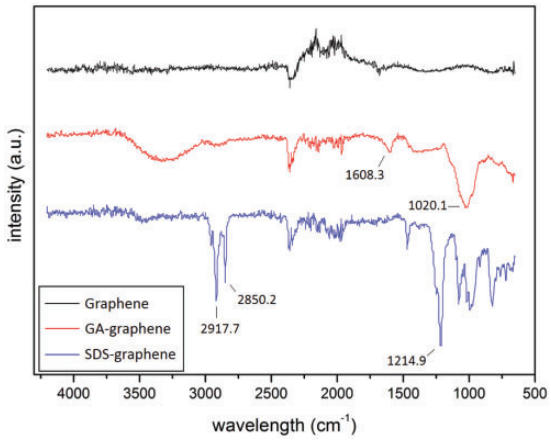


**Figure 2.** FTIR spectrum of modified and unmodified graphene.

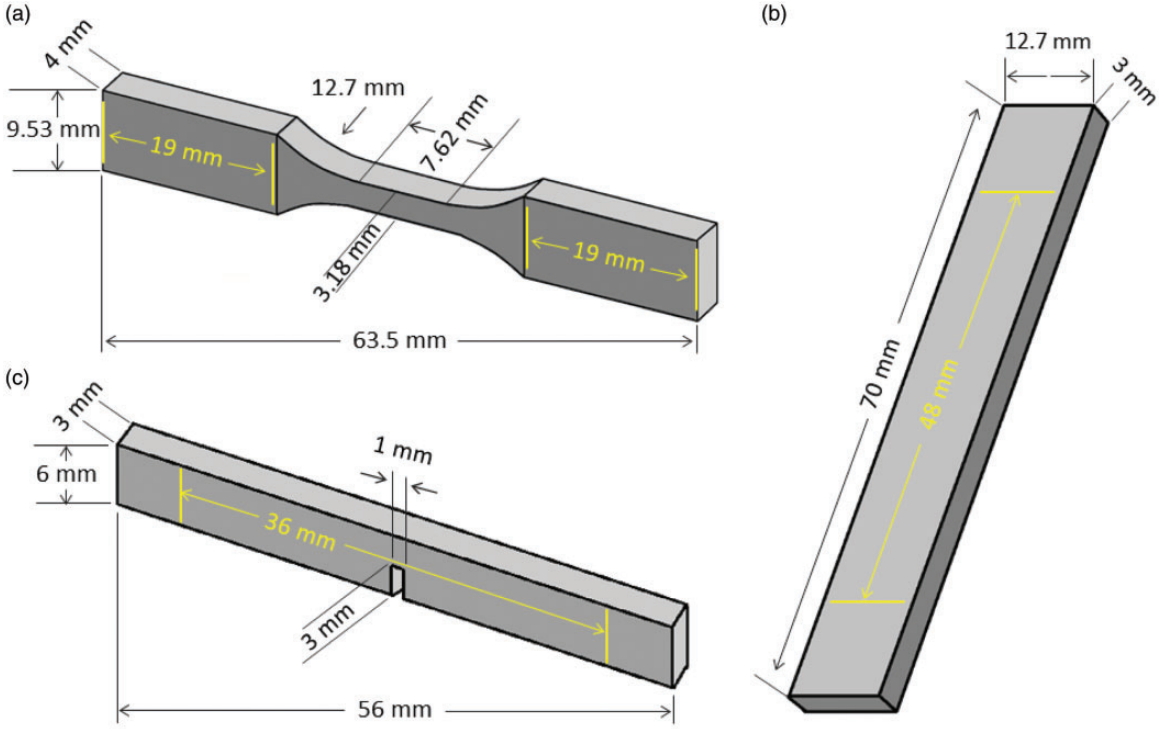


**Figure 1.** Schematics of the testing samples: (a) Tensile; (b) three-point bend; and (c) fracture toughness.

of GA. For the spectrum of SDS–graphene, the two peaks at 2850.2 $cm^{-1}$ and 2917.7 $cm^{-1}$ shows the C–H of the saturated alkyl groups, the peak at 1214.9 $cm^{-1}$ shows the stretching of S=O. Those peaks are the characteristic peaks of SDS and implies the presence of SDS on the graphene surface.

## *Mechanical test*

The uniformly dispersed graphene in the matrix will in return affect the macroscopic properties of the nanocomposites, while most of the work on epoxy/graphene nanocomposites aims at exploiting the mechanical enhancement of the nanocomposites. In this work, tensile test, three-point bend test, fracture test and Vickers hardness test had been carried out and the results are shown in Figure 3(a), (b), (c) and (d) respectively.

As shown in the figure, for all set tests, G-0.3 samples show the lowest properties. After introduced surfactants, all samples show enhanced tensile, flexural, fracture properties and hardness. This improvement is due to that both the surfactants can enhance the graphene dispersion. By comparing the two surfactants, it is obvious that SDS samples show higher values in the properties than those of GA samples, indicating that SDS generates better dispersion than GA.

The improvement in the mechanical properties of nanocomposites can be ascribed to the fact that the dispersion of graphene has been improved. A continuous graphene network can be formed by the uniformly dispersed graphene, thus could release the stress concentration efficiently, and then enhance the mechanical strength and improves the energy-absorbing capacity. Moreover, the uniformly dispersed graphene has changed the microstructure of the polymeric network, which will be discussed in the latter part.

## *TGA test*

Thermal decomposition behaviour is very important for practical application of materials under high temperature; therefore, the TGA curves of nanocomposites had been tested and shown in Figure 4(a). As shown in the figure, a similar two-stage weight loss had been observed for all nanocomposites, means that all nanocomposites had the same decomposition mechanism.

The first derivative of the TGA (DTG) curves is shown in Figure 4(b), it can be seen that all samples showed a wide shoulder peak and a sharp decomposition peak. The shoulder peak corresponds to the first weight loss, which occurred between 100°C and 270°C. This weight loss was caused by the degradation of small molecules on the side chains. The sharp peak

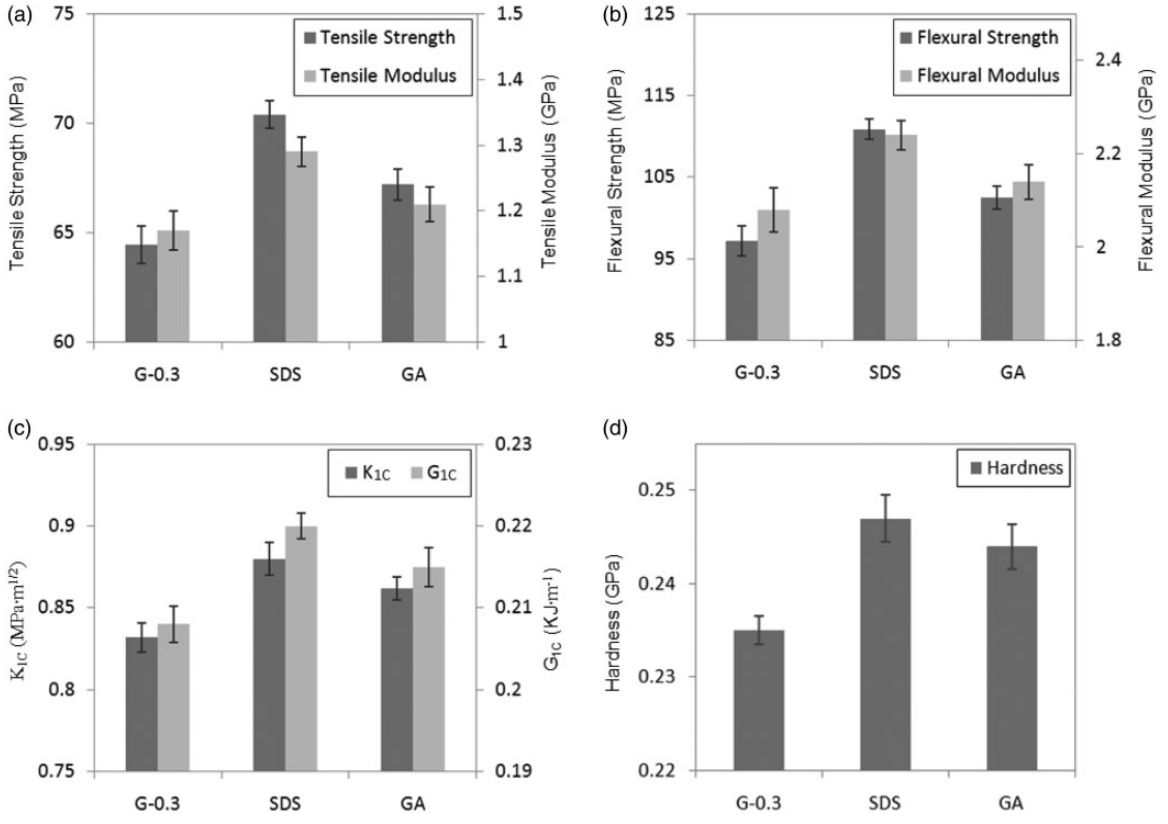


**Figure 3.** Mechanical testing results of the nanocomposites.

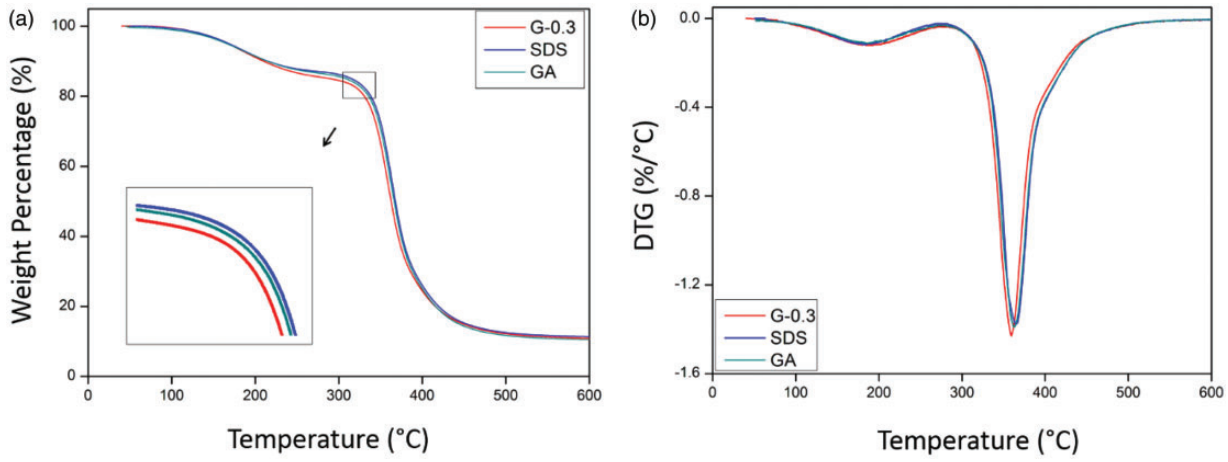


**Figure 4.** TGA and DTG curves of epoxy/graphene nanocomposites.

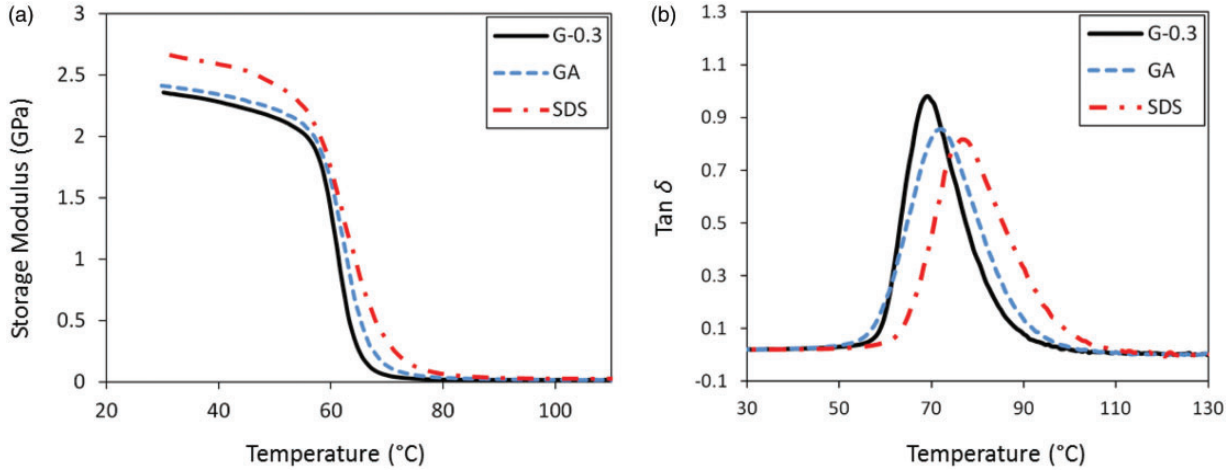


**Figure 5.** DMA testing results. (a) Storage modulus and (b) Tan $\delta$.

on the DTG curve shows the second weight loss occurred between 250°C and 450°C, which was due to the degradation of the main epoxy chain. For the sharp decomposition peak of the material, the peak value features the temperature of maximum decomposition rate, which occurred at 359.42°C for G-0.3 samples. However, this temperature for SDS and GA samples is 366.11°C and 365.02°C, respectively, which showed an improvement in the thermal stability.

The reason for this phenomenon is that the cross-linking density of the epoxy had been increased by the uniformly dispersed graphene. In general, the cross-linked bonds per volume was characterized by the cross-linking density, for typical polymeric materials, high cross-linking density means strong chain bonding, therefore brings the nanocomposites stronger capacity to withstand heat. Compare with GA samples and G-0.3 samples, SDS samples tends to shorten the distance among cross-linking points, and thus increases the cross-linking density of the resultant network. On the other hand, a continuous graphene network had been formed by the uniformly dispersed graphene, this could reduce the volatilization rate of the decomposition products.

In general, the use of SDS resulted in a higher heat capacity of nanocomposites and a better barrier effect of the graphene network. The enhancement in the thermal stability is the result of enhanced dispersion of graphene after introduced SDS.

## *DMA test*

The storage modulus ($E'$) as a function of temperature is shown in Figure 5(a). As in the figure, the storage modulus of samples prepared with surfactants increased obviously over the samples prepared with simple graphene throughout the temperature range investigated. Especially, SDS-prepared samples show

2.71 GPa in the storage modulus, which is higher than that 2.44 GPa of GA samples and 2.35 GPa of G-0.3 samples.

Normally, the peak of tan$\delta$ curve was taken as the glass transition temperature ($T_g$) value, and the segmental motion of polymers is characterized by $T_g$. For G-0.3 samples, the tan $\delta$ peak was observed at 69.28°C as shown in Figure 5(b). For nanocomposites processed by SDS and GA, $T_g$ was observed in higher temperatures. This is due to the phenomenon that graphene had been uniformly dispersed in the matrix had restricted the epoxy chain mobility, and therefore increased the $T_g$ values. Among all the increments, the highest $T_g$ was shown by SDS samples with the value of 76.96°C, which is about 7°C increment compared to the samples prepared with simple graphene, while only slight increases (~3°C) in $T_g$ has been obtained for GA samples. To sum up, the uniformly dispersed graphene can increase the cross-linking density of the epoxy network, and then play a positive role in improving the thermal stability.

### *SEM test*

SEM was used to examine the fracture surfaces of nanocomposites. As shown in Figure 5(a), poorly dispersed graphene is seen on the fractured surface of epoxy, which features a poor interfacial interaction between graphene and the polymer. The poor interface shows the brittle nature of the material. For SDS samples, as shown in Figure 6(b), the fracture surface shows a clear fracture pattern, reveals that the usage of SDS has generated a more uniform graphene dispersion. The homogeneously dispersed graphene is able to bridge growing cracks, impede the crack propagation, and thus lead to enhancement in the properties of the nanocomposites. However, as shown in Figure 6(c), sparsely dispersed graphene aggregates can still be observed on the surface of GA samples. These aggregates act as defects, cause stress concentration and decrease the properties of the nanocomposites.

## Conclusions

A prerequisite for the property enhancement of epoxy/graphene nanocomposites is the uniform dispersion of graphene in the matrix, however, the ultra-high specific surface area of graphene results in high van der Waals forces and thus induces a strong tendency to reaggregate. Therefore, the usage of chemicals to surface modify graphene becomes a very important way to resist this reaggregation.

SDS and GA have been selected to investigate their dispersing effectiveness of graphene in epoxy matrix. The electrostatic repulsions provided by SDS and the steric repulsion provided by GA can resist graphene from reaggregate and result in improved dispersion and homogenous mixing of graphene in epoxy. Mechanical test, DMA, TGA and SEM test have been carried to compare the dispersing effectiveness.

The results show that samples prepared with simple graphene show lowest performance. Non-uniformly dispersed graphene can be seen clearly on the fractured surface of G-0.3 samples. After processed by surfactants, the properties of nanocomposites significantly increased, which means both these two surfactants can produce a fine and homogeneous dispersion of graphene. However, it should be noted that some small aggregation can still be seen on the fractured surface of GA samples, SDS-prepared samples show better mechanical performance and higher $T_g$ than that of GA samples. In general, it is concluded that SDS was found to be more efficient than GA to prepare homogeneous epoxy/graphene nanocomposites.

Meanwhile, SDS and GA are also widely used for the dispersion of other nanomaterials,[40–43] therefore, this paper can not only be referred to epoxy/graphene nanocomposites but also can be referred to other

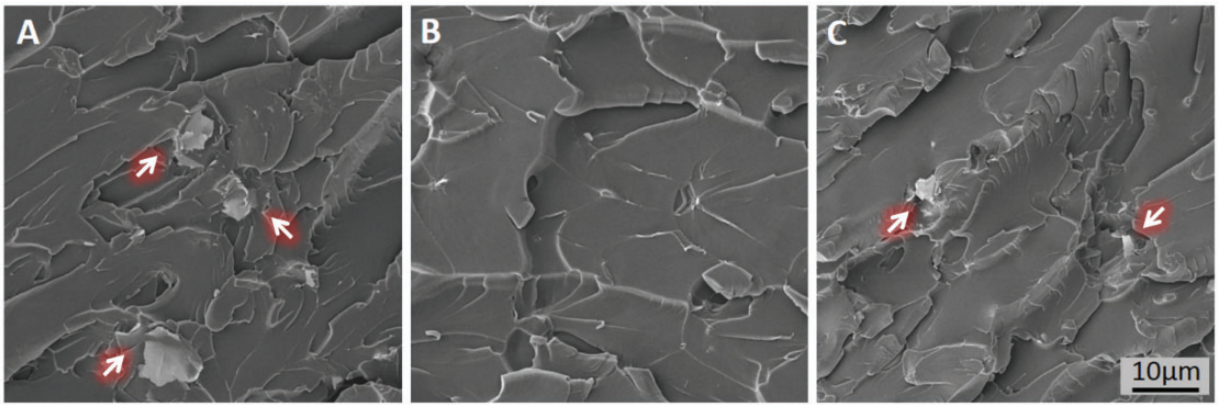


**Figure 6.** SEM images of fracture surfaces of (a) G-0.3 samples; (b) SDS samples and (c) GA samples.

polymer nanocomposites where usage of surfactants is required in the processing.


## Acknowledgement

The authors would like to thank the Department of Mechanical and Construction Engineering, Northumbria University for the provision of research facilities, without which the collection and analysis of relevant data were not possible.


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